# **Solute–Solvent and Solute–Solute Interactions of Resorcinol in Mixed 1,4-Dioxane–Water Systems at Different Temperatures**

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The densities, viscosities, and ultrasonic speeds of resorcinol in 1,4-dioxane + water mixtures and in pure 1,4-dioxane have been measured at 303.15, 313.15, and 323.15 K. Apparent molar volumes  $(V_{\phi})$  and viscosity *B*-coefficients are obtained from these data supplemented with densities and viscosities, respectively. The limiting apparent molar volumes  $(V^0_\phi)$  and experimental slopes  $(S_v^*)$  derived from the Masson equation have been interpreted in terms of solute–solvent and solute–solute interactions, respectively. The viscosity data have been analyzed using the Jones–Dole equation, and the derived parameters *B* and *A* have also been interpreted in terms of solute–solvent and solute–solute interactions, respectively. The structure making/breaking capacities of resorcinol in the studied solvent systems have been discussed. The compressibilities obtained from the data supplemented with their ultrasonic speeds indicate the electrostriction of the solvent molecules around the ions.

**KEY WORDS:** density; electrostriction; mixed aqueous mixtures; resorcinol; solute-solvent and solute–solute interactions; structure maker and breaker; ultrasonic speed; viscosity.

# **1. INTRODUCTION**

Studies on densities, viscosities, and ultrasonic speeds of electrolyte solutions are of great use in characterizing the structure and properties of solutions. Various types of interactions exist between the solutes in solutions, and these solute–solute and solute–solvent interactions are of current interest in all

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branches of chemistry. These interactions provide a better understanding of the nature of the solute and solvent, i.e., whether the solute modifies or distorts the structure of the solvent.

1,4-dioxane and its aqueous mixtures are very important solvents that are widely used in various industries. They figure prominently in the highenergy battery technology and have also found application in organic synthesis as manifested from the physicochemical studies in these media [1–8]. In the present work, an attempt has been made to provide an interpretation of solute–solvent and solute–solute interactions prevailing in the studied electrolyte solutions. Several workers have reported volumetric, viscometric, and ultrasonic studies of this compound in aqueous solutions [9–13], but such studies in pure 1,4-dioxane and its aqueous mixtures are still scarce.

## **2. EXPERIMENTAL**

1,4-dioxane (Merck, India) was kept several days over potassium hydroxide (KOH), refluxed for 24 h, and distilled over lithium aluminium hydride (LiAlH4) as described earlier [1]. Resorcinol (A.R.) was purified by a reported procedure [14], and the compound was dried and stored in a vacuum desiccator. Freshly distilled conductivity water was used.

The densities  $(\rho)$  were measured with an Ostwald–Sprengel type pycnometer having a bulb volume of  $25 \text{ cm}^3$  and an internal diameter of the capillary of about 0.1 cm. The pycnometer was calibrated at 303, 313, and 323 K with doubly distilled water and benzene. The pycnometer with the test solution was equilibrated in a water bath maintained at  $\pm 0.01 \text{ K}$ of the desired temperature by means of a mercury-in-glass thermoregulator, and the absolute temperature was determined by a calibrated platinum resistant thermometer and Muller bridge. The pycnometer was then removed from the thermostatic bath, properly dried, and weighed. The evaporation losses remained insignificant during the time of actual measurements. An average of triplicate measurements was taken into account. The total uncertainty of density is  $\pm 1.0 \times 10^{-5}$  g · cm<sup>-3</sup> and of the temperature is 0.01 K. Details have been described earlier [5, 15, 16].

The viscosities were measured by means of a suspended-level Ubbelohde [17] viscometer at the desired temperature (uncertainty of  $\pm 0.01$  K). The precision of the viscosity measurement was  $\pm 0.003$  mPa · s. Details have been described earlier [5,15,16].

Sound speeds were determined with an uncertainty of  $0.3\%$  using a single-crystal variable-path ultrasonic interferometer (Mittal Enterprises, New Delhi, India) operating at 4 MHz which was calibrated with water, methanol, and benzene at each temperature, as described in detail elsewhere [18, 19]. The experimental values of densities  $(\rho_0)$  and viscosities

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 $(n_0)$  of pure 1,4-dioxane and 1,4-dioxane + water mixtures at 303, 313, and 323 K are reported in Table I. The various salt solutions studied here were prepared by mass, and the conversion of molality to molarity was accomplished [20] using density values.

The experimental values of concentrations  $(c)$ , densities  $(\rho)$ , viscosities  $(\eta)$ , and derived parameters at various temperatures are reported in Table II.

## **3. DISCUSSION**

The apparent molar volumes  $(V_{\phi})$  were determined from the solution densities using the following equation:

$$
V_{\phi} = M/\rho_0 - 1000(\rho - \rho_0)/(c\rho_0)
$$
 (1)

where *M* is the molar mass of the solute,  $c$  is the molarity of the solution, and  $\rho_0$  and  $\rho$  are the densities of the solvent/solvent mixtures and solution, respectively.

The limiting apparent molar volumes  $(V^0_\phi)$  were calculated using a least-squares treatment to the plots of  $V_{\phi}$  versus  $c^{1/2}$  using the following Masson equation [21]:

$$
V_{\phi} = V_{\phi}^0 + S_v^* \sqrt{c} \tag{2}
$$

where  $V^0_{\phi}$  is the partial molar volume at infinite dilution and  $S_v^*$  is the experimental slope. The plots of  $V_{\phi}$  against the square root of the molar concentration  $(c^{1/2})$  were found to be linear with negative slopes. The values of  $V^0_{\phi}$  and  $S^*_{\rm v}$  along with the standard errors are reported in Table III.

As the investigated systems are characterized by hydrogen bonds, the solute–solvent and solute–solute interactions can be interpreted in terms of structural changes, which arise due to hydrogen bond interactions present between various components of the solvent and solution systems.

To examine the solute–solvent interactions,  $V^0_\phi$  can be used. Table III shows that the  $V^0_\phi$  values are positive and increase with a rise in temperature and decrease with an increase in the amount of 1,4-dioxane in the mixtures. This indicates the presence of strong solute–solvent interactions, and these interactions are strengthened with a rise in temperature and weakened with an increase in the amount of 1,4-dioxane in the mixed solvent under investigation, suggesting larger electrostriction at higher temperature and lower amount of 1,4-dioxane in the mixture. Similar results were obtained for some 1:1 electrolytes in aqueous DMF [22] and aqueous THF [1].





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c (mol·dm <sup>-3</sup> )	$\rho$ (g·cm <sup>-3</sup> )	$\eta$ (mPa·s)	$V_{\phi}$ (cm <sup>3</sup> ·mol <sup>-1</sup> )	$(\eta/\eta_0-1)/\sqrt{c}$
10 mass $%$ of 1,4-dioxane + water				
$T = 303.15 K$				
0.0070	1.0077	1.0376	115.9842	$-0.1443$
0.0487	1.0076	1.0255	112.5836	$-0.1066$
0.0905	1.0082	1.0358	103.8412	$-0.0456$
0.1322	1.0091	1.0507	98.9411	0.0012
0.1740	1.0104	1.0789	94.2994	0.0654
0.2157	1.0119	1.1090	90.3421	0.1205
$T = 313.15 K$				
0.0070	1.0012	0.9408	123.5465	$-1.0002$
0.0485	1.0009	0.8334	118.5112	$-0.8534$
0.0901	1.0010	0.8246	112.7892	$-0.6544$
0.1317	1.0019	0.8351	106.0231	$-0.5134$
0.1733	1.0030	0.8620	100.3112	$-0.3845$
0.2150	1.0050	0.9291	92.9054	$-0.2042$
$T = 323.15 K$				
0.0069	0.9975	0.6834	130.0254	$-0.7280$
0.0482	0.9971	0.6939	122.5432	$-0.2092$
0.0895	0.9972	0.7609	115.1204	0.1544
0.1307	0.9979	0.8931	108.6753	0.6304
0.1724	0.9991	1.0793	101.8734	1.1654
0.2140	1.0009	1.2354	94.8752	1.5102
20 mass% of 1,4-dioxane + Water				
$T = 303.15 K$				
0.0071	1.0164	1.2205	114.9854	$-0.0543$
0.0498	1.0163	1.2180	110.8887	$-0.0296$
0.0925	1.0165	1.2265	107.7832	0.0012
0.1352	1.0172	1.2391	102.8712	0.0290
0.1779	1.0187	1.2563	95.8097	0.0584
0.2206	1.0207	1.2699	89.0009	0.0761
$T = 313.15 K$				
0.0071	1.0099	0.9489	118.0543	0.0011
0.0495	1.0098	0.9681	112.9843	0.0914
0.0919	1.0100	0.9939	108.5632	0.1569
0.1343	1.0106	1.0231	104.2871	0.2135
0.1768	1.0118	1.0648	98.7358	0.2906
0.2197	1.0142	1.1113	90.3320	0.3654

**Table II.** Concentration (*c*), Density ( $\rho$ ), Viscosity ( $\eta$ ), Apparent Molar Volume ( $V_{\phi}$ ), and  $(\eta/\eta_0 - 1)/\sqrt{c}$  of Resorcinol in Pure 1,4-Dioxane and 1,4-Dioxane + Water Mixtures at Different Temperatures

$c \text{ (mol} \cdot \text{dm}^{-3})$	$\rho$ (g·cm <sup>-3</sup> )	$\eta$ (mPa·s)	$V_{\phi}$ (cm <sup>3</sup> ·mol <sup>-1</sup> )	$(\eta/\eta_0-1)/\sqrt{c}$
$T = 323.15 K$				
0.0070	1.0035	0.8595	121.0065	$-0.5165$
0.0493	1.0034	0.8189	113.8901	$-0.3984$
0.0912	1.0036	0.8198	109.9987	$-0.2897$
0.1340	1.0041	0.8554	106.0007	$-0.1305$
0.1759	1.0054	0.9046	99.5709	0.0165
0.2179	1.0075	0.9486	91.9964	0.1198
30 mass % of 1,4-dioxane + Water $T = 303.15 \text{ K}$				
0.0070	1.0202	1.4376	103.6557	0.1006
0.0491	1.0208	1.4635	95.5644	0.1200
0.9125	1.0218	1.4805	90.5848	0.1277
0.1334	1.0229	1.4972	87.6753	0.1376
0.1755	1.0241	1.5177	85.6229	0.1544
0.2176	1.0267	1.5326	78.6543	0.1609
$T = 313.15 K$				
0.0070	1.0163	1.2855	105.0988	0.1015
0.0489	1.0168	1.3091	97.6876	0.1223
0.0910	1.0177	1.3260	92.8904	0.1334
0.1328	1.0189	1.3402	88.9876	0.1410
0.1748	1.0202	1.3602	85.9498	0.1605
0.2167	1.0226	1.3787	79.6544	0.1754
$T = 323.15 K$				
0.0070	1.0106	1.1076	106.5169	0.1017
0.0486	1.0111	1.1279	99.6003	0.1222
0.0902	1.01189	1.1445	95.0065	0.1401
0.1318	1.01318	1.1607	89.6543	0.1565
0.1736	1.0143	1.1763	87.8954	0.1704
0.2149	1.0167	1.1899	80.9976	0.1799
Pure 1,4-dioxane				
$T = 303.15 \text{ K}$				
0.0069	1.0203	1.1108	47.4532	0.2540
0.0485	1.0226	1.1514	53.0001	0.2655
0.0902	1.0245	1.1782	57.5543	0.2765
0.1319	1.0261	1.2001	61.8776	0.2841
0.1736	1.0278	1.2222	62.9876	0.2965
0.2152	1.0292	1.2461	65.4432	0.3137

**Table II.** *(Continued)*

c (mol·dm <sup>-3</sup> )	$\rho$ (g·cm <sup>-3</sup> )	$\eta$ (mPa $\cdot$ s)	$V_{\phi}$ (cm <sup>3</sup> ·mol <sup>-1</sup> )	$(\eta/\eta_0-1)/\sqrt{c}$
$T = 313.15 \text{ K}$				
0.0069	1.0147	0.9990	49.4987	0.2544
0.0480	1.0169	1.0362	54.7654	0.2701
0.0894	1.0186	1.0592	60.4765	0.2765
0.1308	1.0202	1.0809	63.8765	0.2901
0.1721	1.0219	1.1041	64.9998	0.3100
0.2134	1.0233	1.1224	66.6279	0.3190
$T = 323.15 \text{ K}$				
0.0068	1.0036	0.8621	50.6542	0.2551
0.0475	1.0058	0.8942	55.9786	0.2709
0.0884	1.0075	0.9146	61.8881	0.2801
0.1293	1.0091	0.9347	64.7831	0.2976
0.1702	1.0107	0.9551	65.8160	0.3176
0.2110	1.0122	0.9738	67.5301	0.3339

**Table II.** *(Continued)*

It is evident from Table III that the  $S_v^*$  values are negative for all temperatures for aqueous mixtures of 1,4-dioxane, but positive for pure 1,4-dioxane. Since  $S_v^*$  is a measure of solute–solute interactions, the results indicate the presence of weak solute–solute interactions. These interactions, however, decrease with a rise in temperature, which is attributed to more violent thermal agitation at higher temperatures, resulting in diminishing the force of solute–solute interactions (ionic dissociation) [7]. The S<sup>∗</sup> values increase with an increase in the amount of 1,4-dioxane in the mixture which results in a decrease in solvation of ions, i.e., more and more solute is accommodated in the void space left in the packing of large associated solvent molecules with the addition of 1,4-dioxane to the mixture.

The variation of  $V_{\phi}^0$  with temperature of resorcinol in solvent mixtures follows the polynomial,

$$
V_{\phi}^{0} = a_0 + a_1 T + a_2 T^2
$$
 (3)

over the temperature range under investigation where *T* is the temperature in K.

Values of coefficients of the above equation for resorcinol for compositions of 1,4-dioxane + water mixtures are reported in Table IV.

From the values of coefficients the following equations are obtained:  $10$  mass  $\%$  1,4-dioxane + water mixture



**Table III.** Limiting Apparent Molar Volume (  $\mathcal{S}^{\bullet}$  $\phi_{\phi}$ ) and Experimental Slope (S\* v $\binom{2}{v}$  for Resorcinol in Different 1,4-Dioxane + Water Mixtures at

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$$
V_{\phi}^{0} = -1123.39/\text{cm}^{3} \cdot \text{mol}^{-1} + 7.23T/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}
$$
  
- 0.010T<sup>2</sup>/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-2} (4)

20 mass % 1,4-dioxane + water mixture

$$
V_{\phi}^{0} = -74.86/\text{cm}^{3} \cdot \text{mol}^{-1} + 1.02T/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}
$$

$$
- 0.001T^{2}/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-2}
$$
(5)

30 mass % 1,4-dioxane + water mixture

$$
V_{\phi}^{0} = -216.27/\text{cm}^{3} \cdot \text{mol}^{-1} + 1.89T/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}
$$

$$
-0.002T^{2}/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-2}
$$
(6)

Pure 1,4-dioxane

$$
V_{\phi}^{0} = -500.30/\text{cm}^{3} \cdot \text{mol}^{-1} + 3.30T/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}
$$
  
- 0.005T<sup>2</sup>/\text{cm}^{3} \cdot \text{mol}^{-1} \cdot \text{K}^{-2} (7)

The apparent molar expansibilities ( $\Phi_{\rm E}^0$ ) can be obtained by the following equation:

$$
\Phi_{\rm E}^0 = (\delta V_\phi^0 / \delta T)_{\rm P} = a_0 + 2a_2 T \tag{8}
$$

The values of  $\Phi_E^0$  of the studied compounds at 303.15, 313.15, and 323.15 K are determined and reported in Table V.

Table IV. Values of Various Coefficients for Resorcinol in Different 1,4-Dioxane + Water Mixtures

Composition of		1,4-dioxane (mass%) $a_0$ (cm <sup>3</sup> · mol <sup>-1</sup> ) $a_1$ (cm <sup>3</sup> · mol <sup>-1</sup> · K <sup>-1</sup> ) $a_2$ (cm <sup>3</sup> · mol <sup>-1</sup> · K <sup>-2</sup> )	
-10	$-1123.39$	7.23	$-0.010$
20	$-74.86$	1.02	$-0.001$
30	$-216.27$	1.89	$-0.002$
100	$-500.30$	3.30	$-0.005$

It is found from Table V that the values of  $\Phi_{\rm E}^0$  decrease with a rise in temperature as well as with an increase in the amount of 1,4-dioxane in the mixture, which can be ascribed to the absence of caging or packing effects [23].

During the past few years it has been emphasized by different workers that  $S_v^*$  is not the sole criterion for determining the structure-making or breaking nature of any solute. Hepler [24] developed a technique of examining the sign of  $(\delta^2 V_\phi^0 / \delta T^2)$  for the solute in terms of long-range structure-making and breaking capacity of the solute in the mixed solvent systems using the general thermodynamic expression,

$$
(\delta c_{\mathbf{P}}/\delta P)_T = -(\delta^2 V_\phi^0/\delta T^2)_{\mathbf{P}}\tag{9}
$$

On the basis of this expression, it has been deduced that the structuremaking solutes should have positive values, whereas structure-breaking solutes should have negative values. In our present investigation, it is evident from Table V that  $(\delta^2 V^0_{\phi}/\delta T^2)$ <sub>P</sub> values are negative for resorcinol and for all 1,4-dioxane + water mixtures investigated here, suggesting thereby that resorcinol acts as a structure breaker in these solvent mixtures.

The viscosity data of solutions for resorcinol in 10, 20, and 30 mass % of 1,4-dioxane + water mixtures and pure 1,4-dioxane have been analyzed using the Jones–Dole [25] equation:

$$
\eta/\eta_0 = 1 + Ac^{1/2} + Bc
$$
\n
$$
(\eta/\eta_0 - 1)/c^{1/2} = A + Bc^{1/2}, \quad \text{where } \eta = (kt - L/t)\rho,
$$
\n(10)

and  $\eta_0$  and  $\eta$  are the viscosities of solvent/solvent mixtures and solution, respectively. k and L are the constants for a particular viscometer, and *t* is

	$\Phi_{\rm F}^0$ (cm <sup>3</sup> ·mol <sup>-1</sup> ·K <sup>-1</sup> )			
Composition of 1,4-dioxane (mass $\%$ )				303.15 K 313.15 K 323.15 K $(\delta \Phi_{\rm F}^0 / \delta T)_P$
10 20 30 100 (Pure)	1.01 0.29 0.25 0.29	0.81 0.27 0.19 0.19	0.59 0.24 0.14 0.09	Negative Negative Negative Negative

**Table V.** Limiting Apparent Molar Expansibilities  $(\Phi_E^0)$  for Resorcinol in Various 1, 4-Dioxane + Water Mixtures at Different Temperatures

the flow time of the solvent/solution in seconds. *A* and *B* are the constants which are estimated by a least-squares method and reported in Table VI.

A perusal of Table VI shows that the values of the *A* coefficient are negative for 10 and 20 mass % of 1,4-dioxane and positive for 30 and 100 mass % of 1,4-dioxane for all temperatures. These results indicate the presence of weak solute–solute interactions, and these interactions increase with an increase of 1,4-dioxane to the mixture.

It is also observed from Table VI that the values of the *B*-coefficient of resorcinol in the studied solvent systems are positive, thereby suggesting the presence of strong solute–solvent interactions, and these types of interactions are strengthened with a rise in temperature and weakened with an increase of 1,4-dioxane in the mixture. These conclusions are in excellent agreement with those drawn from  $V^0_{\phi}$  values discussed earlier.

It has been reported in a number of studies [26, 27] that  $dB/dT$  is a better criterion for determining the structure-making/breaking nature of any solute rather than simply the value of the *B*-coefficient. It is found from Table VI that the values of the *B*-coefficient increase with a rise in temperature (positive  $dB/dT$ ) suggesting the structure-breaking tendency of resorcinol in the solvent systems. A similar result was reported in a study [30] for the case of the viscosity of some salts in propionic acid + ethanol mixtures.

The adiabatic compressibility  $(\beta)$  was calculated from the following relation:

$$
\beta = 1/(u^2 \rho) \tag{11}
$$

where  $\rho$  is the solution density and  $u$  is the sound speed in the solution. The apparent molal adiabatic compressibility ( $\Phi_K$ ) of the liquid solutions was determined from the relation,

$$
\Phi_{\rm K} = M\beta/\rho_0 + 1000(\beta\rho_0 - \beta_0\rho)/(m\rho\rho_0)
$$
\n(12)

The limiting apparent molal adiabatic compressibility ( $\Phi_{\rm K}^0$ ) was obtained by extrapolating the plots of  $\Phi_{\rm K}$  versus the square root of molal concentration of the solute to zero concentration by a least-squares method.

$$
\Phi_{\rm K} = \Phi_{\rm K}^0 + S_{\rm K}^* m^{1/2} \tag{13}
$$

where  $S_K^*$  is the experimental slope.



**Table VI.** Values of

*A* and *B*

Parameters for Resorcinol in Different 1,4-dioxane + Water Mixtures at Different Temperatures





The values of  $u, \beta, \Phi_K, \Phi_K^0$ , and  $S_K^*$  are reported in Table VII.

A perusal of Table VII shows that the  $\Phi_{\rm K}^0$  values increase whereas  $S_K^*$  values decrease with an increase of 1,4-dioxane in the mixtures for the compounds studied here. Since the values of  $\Phi_{\rm K}^0$  and  $S_{\rm K}^*$  are measures of the solute–solvent and solute–solute interactions, respectively, the results are in good agreement with those drawn from the conclusion based on the values of  $\Phi_{\rm K}^0$  and  $S_{\rm K}^*$  explained here earlier. The same result was obtained by us [29] in studies on the sound speeds of some salts in different tetrahydrofuran (THF) + water mixtures.

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